



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

BWSC-CAM

1 May 03

Final

Section V B

Revision No. 3

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Title: Quality Assurance and Quality Control Requirements and Performance Standards
for SW-846 Method 8081A, Chlorinated Pesticides by Gas Chromatography

WSC – CAM – V B

Quality Assurance and Quality Control Requirements for ***SW-846 Method 8081A, Chlorinated Pesticides by Gas Chromatography*** (GC) for the Massachusetts Contingency Plan (MCP)

Document Status: Final

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Document Chronology

| Revision | Status | Date |
|----------|------------------------------|----------------|
| 0 | Public Comment Draft | 5 October 2001 |
| 1 | Revised Public Comment Draft | 17 December 01 |
| 2 | Revised Interim Final | 12 July 2002 |
| 3 | Final | 1 May 2003 |



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for SW-846 Method 8081A, Chlorinated Pesticides by Gas Chromatography

V. Gas Chromatographic (GC) Methods

B. Quality Assurance/Quality Control (QA/QC) Requirements and Performance Standards for SW-846 Method 8081A, Chlorinated Pesticides by Gas Chromatography

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1.0 QA/QC REQUIREMENTS FOR SW-846 METHOD 8081A

1.1 Method Overview

SW-846 Method 8081A is used to determine the concentrations of chlorinated pesticides in extracts from solid and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD) or electrolytic conductivity detectors (ELCD). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target analytes may be determined by either a single- or dual-column chromatographic system. The method also may be applied to other matrices such as oils and wipe samples, if appropriate sample extraction procedures are employed.

1.1.1 Reporting Limits for SW-846 Method 8081A

The reporting limit (RL) using SW-846 Method 8081A for an individual compound is dependent on the concentration of the lowest analytical standard in the initial calibration, choice of sample preparation/introduction method and/or percent (%) solids of the sample. Using standard electron capture detection (ECD), the estimated Reporting Limit (RL) for individual chlorinated pesticides is approximately 1.7-17 µg/kg (wet weight) for soil/sediment samples and 0.05-0.5 µg/L for aqueous samples. Somewhat higher RLs may be expected using electrolytic conductivity detectors (ELCD). No matter which instrument is used, reporting limits for SW-846 Method 8081A will be proportionately higher for sample extracts and samples that require dilution, or when a reduced sample size is used to avoid saturation of the detector.

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for chlorinated pesticides analyzed in support of MCP decision-making are presented in Appendix V B-1 of this document and Appendix VII-A, CAM-VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)".

1.1.2 Additional Requirements

Each laboratory that uses SW-846 Method 8081A is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks as a test of continued performance, and the analysis of laboratory control spikes (LCSs) to assess analytical accuracy. Matrix spikes (MS), matrix spike duplicates (MSD) or Matrix duplicates may also be used to evaluate precision when such samples are analyzed either at discretion of laboratory or at request of data-user.



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Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Section 1.4 and Table V B-1 of this document. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 method 8000B (Section 8.4) and SW-846 method 8081A (Section 8.3). The data associated with the Initial Demonstration of Proficiency should be kept on file at the laboratory and made available to potential data-users on request. The data associated with the Initial Demonstration of Proficiency for SW-846 Method 8081A must include the following:

| QC Element | Performance Criteria |
|-------------------------------|---------------------------------|
| Initial Calibration | CAM-V B, Table V B-1 |
| Continuing Calibration | CAM-V B, Table V B-1 |
| Method Blanks | CAM-V B, Table V B-1 |
| Average Recovery | SW-846 Method 8000, Section 8.4 |
| % Relative Standard Deviation | SW-846 Method 8000, Section 8.4 |
| Surrogate Recovery | CAM-V B, Table V B-1 |
| Internal Standards | CAM-V B, Table V B-1 |

Note: Because of the extensive analyte list and number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all nonconforming analytes. All nonconforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency data provided.

It is essential that laboratory-specific performance criteria for LCS and surrogate recoveries also be calculated and documented. When experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices, the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent quality control performance standards described in Table V B-1 to assess analytical trends (i.e., systematic bias, etc) and improve overall method performance by preempting potential non-conformances.

For SW-846 Method 8081A, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table V B-1. It should be noted that the performance standards listed in Table V B-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory. Laboratories are



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encouraged to continually strive to minimize variability and improve the accuracy and precision of their analytical results.

This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GCs), and skilled in the interpretation of gas chromatograms for individual target and multi-component mixtures of chlorinated pesticides in environmental matrices. Each analyst must demonstrate the ability to produce acceptable quantitative and qualitative results both for individual target and multi-component mixtures of chlorinated pesticides with this method.

1.1.3 Sample Extraction/Cleanup Methods

Sample Extraction and Concentration

Samples are normally prepared by one of the following methods prior to gas chromatographic analysis using SW-846 Method 8081A.

| Matrix | Method |
|---------------|--|
| Air | SW-846 Method 3542 |
| Water | SW-846 Methods 3510C, 3520C, and 3535 |
| Soil/Sediment | SW-846 Methods 3540C, 3541, 3545, and 3550B |
| Waste | SW-846 Methods 3540C, 3541, 3545, 3550B, 3580A, and 3585 |

Extract Cleanup

Extracts may be cleaned up by any of the following methods prior to GC analysis by SW-846 Method 8081A. The recommended cleanup method for routine chlorinated pesticide analyses is SW-846 Method 3660B.

| SW-846 Method | Cleanup Method | Cleanup Type |
|---------------|------------------------------|---------------------|
| 3600 C | General Cleanup Selection | Not Applicable |
| 3610 B | Alumina | Adsorption |
| 3620 B | Florisil | Adsorption |
| 3630 C | Silica Gel | Adsorption |
| 3640 A | Gel-Permeation Cleanup (GPC) | Size-Separation |
| 3660 B | Sulfur Cleanup | Oxidation/Reduction |



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1.2 Summary of Method

A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 C (separatory funnel), Method 3520 C (continuous liquid-liquid extractor), or other appropriate technique.

Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 C (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3546 (microwave extraction), Method 3550 (ultrasonic extraction), Method 3562 (supercritical fluid extraction), or other appropriate technique.

A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Suggested cleanups include alumina (Method 3610), Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660).

After cleanup, the extract is analyzed by injecting a 1 to 2- μ L aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column equipped with either an electron capture or electrolytic conductivity detector.

The chromatographic data produced may then be used to identify and quantify the nineteen individual and multi-component mixtures of chlorinated pesticides listed in Table V B-2.

Identification of chlorinated pesticides based on a single-column analysis must be confirmed on a second column, or must be supported by at least one other independent qualitative technique. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during times of high sample throughput.

Dual column confirmation is not required for samples with concentrations of all individual and multi-component mixtures of chlorinated pesticides below their respective reporting limit.

1.3 Interferences

Refer to SW-846 Methods 3500 (Sec. 3.0, in particular), 3600 C, and 8000 B for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories.



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- Contaminated solvents, reagents, or sample processing hardware,
- Contaminated GC carrier gas, parts, column surfaces, or detector surfaces
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
- Co-elution of target analytes

An in depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

1.3.1 Chemical Contaminants

Major contaminant sources for SW-846 Method 8081A include, but are not limited to, contaminated solvents and inadvertent contact of extraction fluids with rubber and/or plastic materials. The use of non-polytetrafluoroethylene (PTFE) thread sealants or plastic tubing should be avoided. It should be noted that interfering contaminants may also be concentrated during sample preparation and cleanup. Analyses of calibration and reagent blanks provide information about the presence of cross-contamination. When potential interfering peaks are noted in blanks, the analyst should review sample pre-treatment and concentration procedures to identify the source of contamination before re-extraction of the sample.

Raw chromatographic data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem. **Subtracting blank values from sample results is not permitted.** If the laboratory determines that the concentration reported in the blank is so high that false positive results are likely in the associated samples, then the laboratory should fully explain this situation in the case narrative.

Interferences by phthalate esters introduced during sample preparation can pose a major problem in chlorinated pesticide determinations by SW-846 Method 8081A. Common flexible plastics contain varying amounts of phthalate esters, as plasticizers, which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination. These materials may be removed prior to analysis using Method 3640 (Gel Permeation Cleanup) or Method 3630 (Silica Gel Cleanup).



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1.3.2 Cross-Contamination

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of chlorinated pesticide compounds. After the analysis of a sample containing high concentrations of chlorinated pesticide compounds, one or more blanks should be analyzed to check for potential cross-contamination/carryover. The concentration of chlorinated pesticides which can cause cross-contamination/carryover must be determined by the laboratory and will be dependent upon the concentration and level of saturation of the particular analyte. Concentrations of chlorinated pesticide compounds which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample run. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections.

1.3.3 Elemental Sulfur Contamination

The presence of elemental sulfur (S) will result in broad peaks that interfere with the detection of early-eluting chlorinated pesticides. Sulfur contamination should be expected with sediment samples. Sulfur contamination can be removed through the use of SW-846 Method 3660 B.

1.3.4 Co-elution of Target Analytes

As described in Section 3.8 and 3.9 of SW-846 Method 8081A, co-elution among the many target analytes can cause interference problems.

1.3.5 Special Precautions

DDT and endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. The potential for DDT and endrin breakdown should be evaluated before samples are analyzed and at the beginning of each 12-hour shift as described in Section 8.4.6 of SW-846 Method 8081A.

1.4 QA/QC Requirements for SW-846 Method 8081A

1.4.1 General Quality Control Requirements for Determinative Chromatographic Methods

Refer to SW-846 Method 8000B for general quality control procedures for all chromatographic methods, including SW-846 Method 8081A. These requirements ensure that each laboratory



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maintain a formal quality assurance program and records to document the quality of all chromatographic data.

Quality control procedures necessary to evaluate GC system operation may be found in SW-846 Method 8000B, Sec. 7.0, and include evaluation of retention time windows, verification of calibration and chromatographic performance of sample analyses. Instrument quality control and method performance requirements for the GC/ECD system may be found in SW-846 Method 8081A, Sections 8.0 and 9.0, respectively.

1.4.2 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8081A

Specific QA/QC requirements and performance standards for SW-846 Method 8081A are presented in Table V B-1. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide an LSP with "Presumptive Certainty" regarding the usability of analytical data to support MCP decisions. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty", parties must:

- Comply with the procedures described and referenced in CAM-V B;
- Comply with the applicable QC analytical requirements prescribed for the individual testing procedures in the CAM;
- Collect and analyze field QC samples at the frequencies prescribed in the CAM; and
- Adopt the reporting formats and elements specified in the CAM.

In achieving the status of Presumptive Certainty, parties will be assured that analytical data sets:

- ✓ satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data;
- ✓ may be used in a data usability assessment, and, if in compliance with all MCP Analytical Method standards, laboratory QC requirements, and field QC recommended limits and action levels, the data set will be considered usable data to support site characterization decisions made pursuant to the MCP; and



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✓ may be used to support a data representativeness assessment.

1.4.3 Special Considerations for Multi-component Mixtures of Chlorinated Pesticides

The identification of multi-component mixtures (i.e., chlordane) is not based on a single peak, but rather on the characteristic peaks that comprise the "fingerprint" of the mixture, using both the retention times and shapes of the indicator peaks. If, based on site history, multi-component chlorinated pesticides are contaminants of concern, it is the responsibility of the LSP to request that these multi-component analyte spikes be included in the LCSs and MS/MSDs. Multi-component mixtures are not routinely included in LCSs or MS/MSDs.

Table V B-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8081A

| Required QA/QC Parameter | Data Quality Objective | Required Performance Standard | Required Deliverable | Recommended Corrective Action | Analytical Response Action |
|-------------------------------|--------------------------------|--|----------------------|--|--|
| Retention Time Windows | Laboratory Analytical Accuracy | (1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method. (Section 7.6 of SW-846 8000). | No | NA | NA |
| Endrin/DDT Breakdown | Laboratory Analytical Accuracy | (1) Before samples are analyzed and at the beginning of each 12-hour shift. (2) % Breakdown must be ≤ 15 and must be evaluated using peak areas. | Yes | Perform injection port maintenance. Re-calibrate, if required. | Report exceedances in case narrative. |
| Initial Calibration | Laboratory Analytical Accuracy | (1) Minimum of 5 standards. (2) Low standard must be \leq reporting limit. (3) %RSD should be ≤ 20 or "r" should be ≥ 0.99 . (4) Multi-component analytes: Analysis of a single standard at expected mid-point (50%) of calibration range. (5) If regression analysis is used, the curve must not be forced through the origin. (6) Curves must be verified by an independent ICV before analysis. | No | Recalibrate as required by method. | Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the case narrative with a list of the affected analytes. |
| Continuing Calibration (CCAL) | Laboratory Analytical Accuracy | (1) Prior to samples, every 12 hours or every 20 samples, whichever is more frequent, and at the end of the analytical sequence. (2) Concentration level near midpoint of curve. (3) Multi-component analytes should be verified with a one point standard within 12 hrs of being detected in a sample. (4) Percent difference or percent drift of calibration factors should be ≤ 15 . (5) Verify all analytes fall within retention time windows. | No | (1) Perform instrument maintenance, reanalyze CCAL and/or recalibrate as required by method. (2) Reanalyze "associated samples" if beginning or closing CCAL exhibited low response and associated pesticides were or were not detected in samples. (3) Reanalyze "associated samples" if beginning or closing CCAL exhibited high response and associated pesticides were detected in the samples. NOTE: "Associated Samples" refers to all samples analyzed since the last acceptable CCAL. | Report exceedances in case narrative. |

Table V B-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8081A

| Required QA/QC Parameter | Data Quality Objective | Required Performance Standard | Required Deliverable | Recommended Corrective Action | Analytical Response Action |
|---|---|--|------------------------------------|---|--|
| Method Blanks | Laboratory Method Sensitivity (contamination evaluation) | (1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be less than or equal to reporting limit. | Yes | Locate source of contamination; correct problem; re-extract associated samples if contaminants are present in the method blank. | (1) Report nonconformances in case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention (such as the case narrative), should qualify the sample results. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction. |
| Laboratory Control Spikes ¹ (LCSs) | Laboratory Method Accuracy | (1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level should be between low and mid-level standard. (4) Must contain all single-component target analytes. (5) Matrix-specific (e.g., soil, water). (6) Percent recoveries must be between 40-140. (7) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above. | Yes | Recalculate the percent recoveries. Check MS/MSD; if recoveries are acceptable in MS/MSD, nonconformance may be isolated to LCS. If recoveries are outside criteria in MS/MSD, re-extract associated samples. | (1) Report exceedances in case narrative. (2) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (3) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction. |
| MS/MSDs ¹ | Method Accuracy in Sample Matrix Method Precision in Sample Matrix | (1) Extracted with every 20 samples at discretion of laboratory or at request of the data user. (2) Matrix-specific. (3) Prepared using standard source different than that used for initial calibration. (4) Concentration level should be between low and mid-level standard. (5) Must contain all single-component target analytes. (6) Percent recoveries should be between 30-150. (7) RPDs should be ≤30 for single-component analytes and ≤50 for multi-component analytes. | Yes When requested by data user | Check LCS; if recoveries acceptable in LCS, evaluate alternate cleanup techniques for samples associated with MS/MSD and or narrate non-conformance. | Report exceedances in case narrative. |

Table V B-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8081A

| Required QA/QC Parameter | Data Quality Objective | Required Performance Standard | Required Deliverable | Recommended Corrective Action | Analytical Response Action |
|----------------------------------|---|---|--|---|--|
| Surrogates | Accuracy in Sample Matrix | (1) Minimum of 2, one that elutes at beginning of GC run and one that elutes at end of GC run. Recommended surrogates: TCMX and DCB (2) Percent recoveries must be between 30-150 for both surrogates on both columns. (3) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above. | Yes (report surrogate recoveries from both columns) | If the same surrogate is outside limits on both columns, re-extract the sample. If both surrogates are outside limits on one column only, reanalyze the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. | (1) Report exceedances in case narrative. (2) If re-extraction or reanalysis yields similar surrogate non-conformances, the laboratory should report results of both extractions or analyses. (3) If re-extraction or reanalysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction or reanalysis only. (4) If re-extraction or reanalysis is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial and re-extraction or reanalysis. (5) If sample is not re-extracted or reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report. |
| Internal Standards (Optional) | Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix | (1) Minimum of 1. (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ± 30 seconds of retention times in associated continuing calibration standard | No | If internal standard is outside limits, reanalyze sample unless obvious interference present. | (1) Report exceedances in case narrative. (2) If reanalysis yields similar internal standard nonconformance, the laboratory should report both results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recovery, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of holding time and yields acceptable internal standard recovery, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report. |

Table V B-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8081A

| Required QA/QC Parameter | Data Quality Objective | Required Performance Standard | Required Deliverable | Recommended Corrective Action | Analytical Response Action |
|---------------------------------|------------------------------|---|----------------------|-------------------------------|---|
| Identification and Quantitation | Inter-laboratory consistency | (1) Laboratory should use the average calibration factor of each single-component analyte for quantitation. (2) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive pesticide results. The laboratory must report the higher of the two results. All required QA/QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (3) Multi-component analytes: Quantitate as per method requirements (Section 7.6 of SW-846 8081A). | No | NA | (1) If the RPD between the dual column results exceeds 40, the laboratory should qualify the sample results and/or note the exceedance in the case narrative. NOTE: If the high RPD can be definitively attributed to interference on one of the two columns, the laboratory should report the lower value and provide a discussion in the case narrative that this approach was employed. (2) If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the case narrative with a list of the affected analytes. |
| General Reporting Issues | NA | (1) The laboratory must report values \geq the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the <u>lowest</u> dilution within the valid calibration range for <u>each</u> analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. NOTE: Laboratories shall not perform dilutions on samples due to sulfur interference. Laboratories must employ a cleanup technique to reduce the presence of sulfur interference. | Yes | NA | (1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) The performance of dilutions must be documented in the case narrative |

1. Refer to Section 1.4.3 for guidance regarding the inclusion of multiple-component chlorinated pesticide mixtures in LCSs and MS/MSDs

GC = Gas Chromatography

MS/MSDs = Matrix Spikes/Matrix Spike Duplicates

%RSD = Percent Relative Standard Deviation

DCB = Decachlorobiphenyl

"r" = Correlation Coefficient

RPDs = Relative Percent Differences

TCMX = Tetrachloro-m-xylene

ICV = Initial Calibration Verification – separate source standard



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1.5 MCP Analyte List for SW-846 Method 8081A

The MCP analyte list for SW-846 Method 8081A, presented in Table V B-2, is intended to be protective of human health and the environment. The list is comprised of potential contaminants that are readily-analyzable by SW-846 Method 8081A and have compound-specific MCP Method 1 Groundwater/Soil Standards as described in 310 CMR 40.0974 and 40.0980, respectively. In addition, a limited number of chlorinated pesticides that are occasionally encountered during MCP site assessments are also included on the SW-846 Method 8081A analyte list (Consensus Contaminants). These contaminants have Reportable Concentrations (RCs) as described in 310 CMR 40.0360 and 40.1600 and published EPA Integrated Risk Information System (IRIS) toxicity values. Analytes without a promulgated MCP Method 1 Standard have sufficient toxicity information for the Department (or others) to develop compound-specific MCP Method 2 Standards for groundwaters and soils, as described in 310 CMR 40.0980.

The MCP Method 1 Groundwater/Soil Standards used to characterize the risk of harm posed by oil or hazardous materials at a disposal site are described in 310 CMR 40.0974(2), Table 1. This list of groundwater/soil standards, developed by the Department, takes into account a defined set of conservative potential exposure pathways likely to be encountered at most disposal sites. Method 1 Standards have been developed by the Department for over one hundred organic and inorganic contaminants that are commonly encountered at disposal sites. The MCP Method 1 Groundwater/Soil Standards list is periodically reviewed and updated by the Department. When compounds are added to the MCP Method 1 Groundwater/Soil Standards list that are suitable for analysis by SW-846 Method 8270C, the analyte list for this method will be updated accordingly.

MCP Method 2 Groundwater/Soil Standards are developed by the Department (or others) for contaminants of concern for which MCP Method 1 Standards have not been promulgated. The use of Department-developed MCP Method 2 Standards is discretionary. Alternatively, site-specific MCP Method 2 Standards may be developed or a Method 3 risk characterization, as described in 310 CMR 40.0990, may be conducted to evaluate or characterize the risk of harm posed by oil or hazardous materials at a disposal site.

1.6 Additional Reporting Requirements for SW-846 Method 8081A

While it is not necessary to request and report all the SW-846 Method 8081A analytes listed in Table V B-2 to obtain Presumptive Certainty, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830



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In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- Uncharacterized sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or
- Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note that a desire to avoid detection and quantitation of a contaminant that is present or likely present at a site above background levels is not a valid reason to limit an analyte list, and that such an action could constitute a criminal violation of MGL c. 21E.

In cases where a truncated list of method analytes is selected, laboratories must still employ the method-specific quality control requirements and performance standards associated with the requested analytes list to obtain Presumptive Certainty status.

The Reporting Limit (based on the concentration of the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Method 1 Standards, RfDs, benchmark values, background, etc.) with the exceptions footnoted in Table V B-2. Meeting “MCP program” reporting limits may require analytical modifications, such as increased sampling weight or volume to increase sensitivity. All such modifications must be described in the Environmental Laboratory case narrative.



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| Analyte | CASRN | MCP CLEANUP STANDARDS | |
|---|------------|-----------------------|-----------------|
| | | GW-1 | S-1/GW -1 |
| | | µg/L (ppb) | µg/g (ppm) |
| Aldrin | 309-00-2 | 0.5 | 0.03 |
| α-BHC | 319-84-6 | X ¹ | X ¹ |
| β-BHC | 319-85-7 | X ¹ | X ¹ |
| γ-BHC (Lindane) | 58-89-9 | 0.2 | 0.1 |
| δ -BHC | 319-86-8 | X ¹ | X ¹ |
| Chlordane (nos), <i>multi-component mixture</i> | 57-74-9 | 2 | 1 |
| 4,4' -DDD | 72-54-8 | 0.1 | 2 |
| 4,4'-DDE | 72-55-9 | 0.1 | 2 |
| 4,4' -DDT | 50-29-3 | 0.3 | 2 |
| Dieldrin | 60-57-1 | 0.1 | 0.03 |
| Endosulfan I ² | 959-98-8 | 0.1 (GW-3) | 0.05 (S-1/GW-3) |
| Endosulfan II ² | 33213-65-9 | 0.1 | 0.05 |
| Endosulfan Sulfate | 1031-07-8 | X ¹ | X ¹ |
| Endrin | 72-20-8 | 2 | 0.6 |
| Endrin ketone | 53494-70-5 | X ¹ | X ¹ |
| Heptachlor | 76-44-8 | 0.4 | 0.1 |
| Heptachlor epoxide | 1024-57-3 | 0.2 | 0.06 |
| Hexachlorobenzene | 118-74-1 | 1 | 0.7 |
| Methoxychlor | 72-43-5 | 2 (GW-3) | 30 (S-1/GW-3) |

(nos) – not otherwise specified

1 Department-Developed MCP Method 2 Standard. Use of these Standards is discretionary

2 One of two isomers that comprise Endosulfan, CAS Number 115-29-7. Total concentration of both isomers must be used to evaluate compliance with MCP Method 1 Standards.



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2.0 Data Usability Assessment for SW-846 Method 8081A

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for SW-846 Method 8081A which may be used to assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document

3.0 Reporting Requirements for SW-846 Method 8081A

3.1 General Reporting Requirements for SW-846 Method 8081A

General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in CAM-VII A, Section 2.4. This guidance document provides recommendations for field QC, as well as the required content of the Environmental Laboratory Report, including:

- Laboratory identification information presented in CAM-VII A, Section 2.4.1,
- Analytical results and supporting information in CAM-VII A, Section 2.4.2,
- Sample- and batch-specific QC information in CAM-VII A, Section 2.4.3,
- Laboratory Report Certification Statement in CAM-VII A, Section 2.4.4,
- Copy of the Analytical Report Certification Form in CAM-VII A, Exhibit VII A-1,
- Environmental Laboratory Case Narrative contents in CAM-VII A, Section 2.4.5,
- Chain of Custody Form requirements in CAM-VII A, Section 2.4.6

3.2 Specific Reporting Requirements for SW-846 Method 8081A

Specific Quality Control Requirements and Performance Standards for SW-846 Method 8081A are presented in Table V B-1. Specific reporting requirements for SW-846 Method 8081A are summarized below in Table V B-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements should always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data are to be available for review during an audit and may also be requested on a client-specific basis.



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Table V B-3 Routine Analytical Reporting Requirements for SW-846 Method 8081A

| Parameter | Required Analytical Deliverable |
|-------------------------------------|--|
| Retention Time Windows | NO |
| Endrin/DDT Breakdown Check Standard | YES |
| Initial Calibration | NO |
| Continuing Calibration (CCAL) | NO |
| Method (Preparation) Blank | YES |
| Laboratory Control Spikes (LCSs) | YES |
| Matrix Spike (MS) | YES (if requested field MS) |
| Matrix Spike Duplicate (MSD) | YES (if requested field MS/MSD) |
| Matrix Duplicate (MD) | YES (if requested by Data User) |
| Surrogates | YES |
| Internal Standards (ISs) | NO |
| Identification and Quantification | NO |
| General Reporting Issues | YES |



Title: Sample Preservation, Container And Analytical Holding Time Specifications For Surface Water, Groundwater, Soil, And Sediment Matrices For Chlorinated Pesticides Analyzed In Support Of MCP Decision-Making

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for chlorinated pesticides analyzed in support of MCP decision-making are summarized below and presented in Appendix VII-A of CAM-VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)".

| Matrix | Container ¹ | Preservation | Holding Time ² |
|--|--|---|--|
| Aqueous Samples, with no Residual Chlorine | (2) 1-L amber glass bottles w/ Teflon-lined screw caps | Cool to 4°C | 7 days to extraction; 40 days from extraction to analysis |
| Aqueous Samples, with Residual Chlorine ³ | (2) 1-L amber glass bottles w/ Teflon-lined screw caps | Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) ⁴ . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4°C | 7 days to extraction; 40 days from extraction to analysis |
| Soil/Sediment Samples | (1) 8-oz. amber glass jar w/ a Teflon-lined screw cap | Cool to 4°C | 14 days to extraction; 40 days from extraction to analysis |
| Waste Samples | (1) 500 mL amber wide mouth jar with a teflon lined screw cap. | No special preservation required | 14 days to extraction; 40 days from extraction to analysis |

1 The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis

2 Holding time begins from time of sample collection.

3 Presence of residual chlorine is usually associated with drinking water samples

4 Confirm dechlorination. If Residual Chlorine > 5 mg/L additional dechlorination agent may be required



Title: Summary of Minimum Field QC Sample Frequency in Support of “Presumptive Certainty” Status for SW-846 Method 8081A, Chlorinated Pesticides by Gas

Parties seeking “Presumptive Certainty” for chlorinated pesticide analyses by SW-846 Method 8081A are required to provide the laboratory with the appropriate volumes and types of quality control (QC) samples as summarized below. Except for samples that require prior laboratory knowledge (i.e., Matrix Spikes, Matrix Spike Duplicates, drinking water samples, trip blanks/equipment blanks), field QC samples must be submitted to the laboratory as blind samples, using the containers and preservatives specified in CAM-VII A, Appendix VII A-1, and Appendix V B-1 of this document.

| ANALYTES | METHOD | QC ELEMENT | MEDIA | | |
|------------------------|---------------------|-------------------|----------------------------|----------------------------|----------------------------------|
| | | | SOIL/SEDIMENT | AQUEOUS | DRINKING WATER |
| Chlorinated Pesticides | SW-846 Method 8081A | Matrix Spike (MS) | Not Mandatory ^a | Not Mandatory ^a | Not Mandatory ^a |
| | | Field Duplicate | Not Mandatory ^a | Not Mandatory ^a | If analyte detected ^b |
| | | Trip Blank | Not Mandatory ^a | Not Mandatory ^a | Not Mandatory ^a |

a Samples designated “Not Mandatory” are not needed for “Presumptive Certainty” acceptance of analytical data. However, on a site and project-specific basis, the use of one or more of these and other QC elements (e.g. equipment rinsate blanks, etc.) may be advisable and/or necessary to demonstrate usability of the data, and/or to determine if the data are biased high due to contamination by sampling equipment/storage conditions.

b A duplicate analysis MUST be conducted for any drinking water sample if one or more analytes are detected above the reporting limit (RL). The laboratory must be provided with adequate sample volume to satisfy this contingency. Either a duplicate sample or other means may be used to assure that the laboratory has sufficient sample volume to re-extract and reanalyze the sample.